

REMARKS

Claims 1 - 10 and 21 - 23 remain active in this application. Claims 11 - 20 have previously been canceled. Claim 10 remains withdrawn from consideration. The indication of allowability of claims 8, 9 and 22 is noted with appreciation. Amendment of claim 23 has been requested to change dependency to allowed claim 22 as noted in the remarks filed January 18, 2007. No new matter has been introduced into the application.

Claims 1 - 9, 21 and 23 have been rejected under 35 U.S.C. §112, first paragraph, as failing to comply with the written description requirement and under 35 U.S.C. §112, second paragraph as being indefinite; the Examiner asserting that the phrase "stoichiometric alloy" in amendatory language presented in the response filed January 18, 2007, has no established meaning and is not sufficiently defined in the specification and criticized antecedent language correspondence in claim 23. These grounds of rejection are respectfully traversed.

It is respectfully submitted that the Examiner's criticism of claim 23 is now moot in view of the amendment of dependency requested above. Dependency of claim 23 has been changed to depend from claim 22 in which ample and exact antecedent language is present.

In regard to the Examiner's criticism of amendatory language, as pointed out in the response filed January 18, 2007, substantially verbatim support for the amendatory language presented therein is found in paragraph [0018] of the specification as originally filed. While the Examiner is substantially correct in asserting that the term "stoichiometric" is commonly used to refer to the fixed, usually rational, numerical relationship between quantities of substances

participating in a chemical reaction or constituting a compound (see, for example, the Oxford English Dictionary), the formation of an alloy differs from chemical reactions but is no less a "reaction" than chemical reactions where atoms are bound together accompanied by a change in physical and chemical properties of the resulting material and, in fact, the phrase "reaction front" is used in the same sentence where "stoichiometric alloy" appears in paragraph [0018]. Moreover, chemical compounds can be either stoichiometric or non-stoichiometric as noted on the attached page 14 of a standard textbook "Chemical Principles and Properties" by Sienko and Plane (McGraw-Hill, 1974).

The phrase "stoichiometric alloy" is well-understood and accepted in the art (see, for example, the attached article "Stoichiometric and Off-Stoichiometric Alloying in Silicide Compound Ti_5Si_3 By Nb or Cr Additions" by Zhang et al., Mat. Res. Soc. Symp. Proc., Vol. 552 (1999), copy attached) to indicate an alloy having no excess of any constituent material such as where each constituent material is saturated with the other constituent materials in solid solution and the atomic proportions of at least some constituent materials or combinations of materials is an exact ratio. Such a stoichiometric alloy is necessarily formed at an interface or "reaction front" of copper and an alloying material, such as those disclosed in paragraph [0025] of the specification as originally filed, in an amount equal to or in excess of the amount reacted with the copper. Paragraph 0025 also refers to "reaction" of the alloying material and copper as well as the removal of excess unreacted alloying material after high temperature deposition which cause the alloying reaction as the alloying material is deposited and diffused into (also

fully supporting the recitation of "continuously graded") the copper. The plain fact of the matter is that, while many alloys will be non-stoichiometric and most compounds are stoichiometric, an alloy in which the alloying materials precisely fill a crystal lattice in a regular pattern consistent with each material being saturated with a solid solution of other materials will necessarily be stoichiometric in accordance with the same definition applied to compounds.

Therefore, it is respectfully submitted that the Examiner's position in regard to this ground of rejection is untenable since it necessarily contradicts the explicit disclosure which unequivocally states that a stoichiometric alloy is formed at the reaction front where alloying takes place. These grounds of rejection are completely grounded in the Examiner's assertion, contrary to the disclosure and without documentation, that no stoichiometric alloys can exist and that no "reaction" is involved in their formation neither of which is correct. The amendatory language of the claims simply recites the "reaction front where a stoichiometric alloy has been formed" as a *structural element* of the claimed integrated circuit; which structural element is well-supported by the explicit language of the specification as originally filed. The term "stoichiometric" is used precisely in accordance with the generally accepted definition thereof and no additional definition is needed or appropriate. Accordingly, reconsideration and withdrawal of these grounds of rejection are clearly in order and are respectfully requested.

Claims 1 - 4, 6 and 7 have been rejected under 35 U.S.C. §103 as being unpatentable over Besser in view of Lopatin and claim 5 has been rejected under 35 U.S.C.

§103 as being unpatentable over Besser in view of Lopatin and Wang. These grounds of rejection are again respectfully traversed for the reasons of record and the further remarks provided below.

The Examiner asserts that claim 1 and claims 2 - 9 and 21, depending therefrom, are product-by-process claims and thus does not address the amendatory language previously submitted. It is respectfully submitted, as alluded to above, that there is no process recitation contained in claim 1 and that the amendatory language clearly and explicitly recites structural features. Specifically, the "reaction front where a stoichiometric alloy is formed" is clearly a recitation of a *location* from which the alloying material concentration is graded. Likewise, the recitation of the alloying material "*being*" fully reacted with the metal is a structural recitation of the composition of claimed structure including the substantial absence of unreacted alloying material and clearly not a process recitation. Therefore, the product-by-process construction of the claims by the Examiner is respectfully submitted to be wholly unwarranted and improper and certainly does not excuse the failure by the Examiner to address all recitations of the claims or to make a *prima facie* demonstration of obviousness of any claim in the application.

Similarly, the failure of the Examiner to address all recitations of the claims or to make a *prima facie* demonstration of obviousness of any claim in the application is not excused by the Examiner's improper attempt to buttress these grounds of rejection with improper grounds of rejection under 35 U.S.C. §112 in which the Examiner merely asserts, without any evidence whatsoever, the impossibility of the subject matter actually claimed. It is respectfully submitted that such

an assertion is a strong indication of the *unobviousness* of the claimed subject matter and, in any event, any rejection based on prior art must present and be based on *evidence*, whether of the scope and content of the prior art or at least in support of whatever it is that the Examiner asserts. In this case, the Examiner has not addressed explicit structural recitations of the claims which the Examiner improperly seeks to justify by an improper and unjustified assertions that claims 1 - 9 and 21 are product-by-process claims as well as by improperly and erroneously asserting a deficiency of the written description and indefiniteness of the claims; neither assertion being based on any discernible evidence, either in the claims or the prior art.

Therefore, it is respectfully submitted that these grounds of rejection under 35 U.S.C. §103 are clearly in error and untenable. Accordingly it is respectfully submitted that reconsideration and withdrawal of these grounds of rejection are in order and such action is respectfully requested.

It is also respectfully submitted that the finality of the present official action is premature. No action can properly be made final if the action does not *prima facie* establish the propriety of grounds of rejection contained therein. Therefore, it is respectfully requested that the finality of the present action be withdrawn and the above-requested amendment entered as a matter of right.

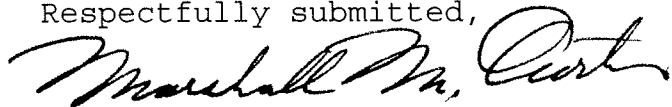
In any case, it is also respectfully submitted that entry of the above-requested amendment is well-justified. The requested amendment is limited to a change of dependency in response to a criticism by the Examiner and, as such, cannot raise a new issue. Further, the proper dependency of claim 23 is readily determinable

from the remarks of the amendment filed January 18, 2007, to which the above-requested amendment conforms. Moreover, the amendment clearly places the application in condition for allowance or reduces issues and improves form for appeal. Accordingly, entry of the above amendment is respectfully requested.

Since all rejections, objections and requirements contained in the outstanding official action have been fully answered and shown to be in error, it is respectfully submitted that reconsideration is now in order under the provisions of 37 C.F.R. §1.111(b) and such reconsideration is respectfully requested. Upon reconsideration, it is also respectfully submitted that this application is in condition for allowance and such action is therefore respectfully requested.

If an extension of time is required for this response to be considered as being timely filed, a conditional petition is hereby made for such extension of time. Please charge any deficiencies in fees and credit any overpayment of fees to Attorney's Deposit Account No. 50-2041.

Respectfully submitted,



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Attachments:

Chemical Principles and Properties, Sienko & Plane, p.14
Zheng et al.: "Stoichiometric and Off-Stoichiometric
Alloying..."

as the atomic weight of scandium, not very far from the approximate value of 47 amu guessed from the law of Dulong and Petit.

The procedure outlined above for getting at the atomic weight is satisfactory as long as we can be absolutely certain that the ratio of atoms of oxygen to atoms of scandium is precisely 3:2. In other words, we have to assume that the atomic ratio is rational and simple. If we believe in undivided atoms, then the atomic ratio must be expressible as the ratio of whole numbers. Dalton argued further that these whole numbers must be small ones; so atomic ratios can only be very simple, for example, 1:1, 1:2, and 2:3. Such compounds, in which atomic ratios are fixed by ratios of small whole numbers, are called *stoichiometric*.^{*} If the ratios are not fixed at simple values, the compounds are referred to as *nonstoichiometric*; they "deviate from stoichiometry." As an example, whereas ordinary stoichiometric scandium oxide has an oxygen-to-scandium ratio of 3:2, it is possible by going to very high temperatures to prepare a nonstoichiometric scandium oxide where the oxygen-to-scandium ratio is, for example, 1.496. Nonstoichiometric compounds generally have unusual and interesting electric properties; they have recently become the object of intensive study. It is amusing to note that John Dalton had a rival in his day, Claude Berthollet, who argued vehemently that compounds did not have to be stoichiometric. Berthollet lost the argument then, but the controversy is memorialized in modern nomenclature, where nonstoichiometric compounds are called *berthollides* and stoichiometric compounds are called *daltonides*.

Modern procedure for determining atomic weights does not depend on chemical combining weights and on guesses of atomic ratios but relies on direct determination of the absolute masses of isotopes and the counting of their relative abundances. The device used is called a *mass spectrometer*. Its operation will be understood better after consideration of Sec. 1.5, but the principle of operation is to whirl atoms in spiral tracks so that the more massive ones tend to collect on the outside because of their greater inertia to being deviated from straight-line motion. A beam of atoms composed of several isotopes can thus be split into several beams, each composed of only one isotope. From the positions of the beams and their relative intensities one can deduce the masses and the number of atoms of each type. The following example shows how mass spectrometric data can be used to calculate the atomic weight of an element.

Example 1.1

Natural chlorine consists of a mixture of two isotopes, designated, respectively, as chlorine 35 and chlorine 37. Mass spectrometric analysis indicates that 75.53% of the atoms are chlorine 35, having a mass of 34.968 amu, and 24.47% are chlorine 37, having a mass of 36.956 amu. Calculate the chemical atomic weight of natural chlorine.

^{*} The first syllable *stoi* is pronounced *stoy* (rhymes with *boy*); it is often mispronounced *stow*. Stoichiometry comes from the Greek *stoicheion*, meaning "element" and *metron* meaning "measure."

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The illustration on the cover is a photomicrograph of a silicon-carbide crystal developed by vapor-phase condensation. The spiral pattern of contour lines show the spiral pattern of crystal growth. The "rainbow" lines indicate crystal growth along lines of stress. The vari-colored patches at left bottom and right mark impurities in the crystal. Silicon carbide is used extensively in heavy-duty industrial abrasives. The magnification is approximately 1400 diameters. The micrograph was made by the late F. Hubbard Horn and is shown here through the courtesy of the General Electric Research and Development Center, Schenectady, New York.

Preface

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Stoichiometric and Off-Stoichiometric Alloying in Silicide Compound Ti_5Si_3 By Nb or Cr Additions

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ABSTRACT

Titanium silicide Ti_5Si_3 , whose melting temperature is 2130°C bears the potential for very high temperature application. This paper reports our results on the alloying behaviour of Nb or Cr addition to this compound. Total energy calculation shows that the substitution of Ti by Nb or Cr atoms in Ti_5Si_3 crystal stiffens the bonding between the atoms. In experiment, two means of alloying are considered: stoichiometric and off-stoichiometric alloying. Stoichiometric alloying in Ti_5Si_3 results in compounds consisting of single Ti_5Si_3 phase while off-stoichiometric alloying yields hypereutectic microstructure with Ti_5Si_3 being the primary phase. The Ti_5Si_3 phase in both cases dissolves certain amount of Nb or Cr alloying element and its composition agrees with the stoichiometric composition of $(\text{Ti,Nb})_5\text{Si}_3$ or $(\text{Ti,Cr})_5\text{Si}_3$. The moduli of the stoichiometric alloys increase with the increase of alloying element addition, indicating an enhancement in Ti_5Si_3 crystal.

INTRODUCTION

A need for high performance materials at temperatures up to 1600°C leads to the interest in the study of silicon compounds. Compared with the aluminide materials, silicides have the advantages of higher melting points, lighter weight and better oxidation resistance, etc. Among them, Ti_5Si_3 is expected to be a potential candidate for very-high-temperature structural application due to its high melting temperature (2130°C) and low density (4.32 g/cm^3). G. Frommeyer is one of the first researchers who carried out investigations to evaluate the basic properties of Ti_5Si_3 intermetallic compound [1]. In the years followed, a few studies on the physical and mechanical properties of Ti_5Si_3 were reported by other researchers [2-5]. Still, there is much left for us to know about Ti_5Si_3 . For instance, the alloying behaviour of Ti_5Si_3 remains unreported, since most of the previous works dealt with the binary Ti_5Si_3 . Alloying addition to Ti_5Si_3 is a possible way to improve the properties of this compound. In this paper, the alloying behaviour of Ti_5Si_3 with Nb and Cr additions was carefully investigated through two ways of alloy design in order to gain a further understanding in this compound.

THEORETICAL CALCULATION

Theoretical calculation of the alloying effect in Ti_5Si_3 compound is based on the ideal cleavage model in which the crystal cell is divided into two halves along a selected crystallographic plane (hkl), i.e. the cleavage plane. By increasing the inter-planar spacing between the two cleavage planes, total energy is calculated as a function of the inter-planar spacing. Thus cleavage energy is expressed as:

$$E_{\text{cleavage}} = E_{\text{ideal}} - E_{\infty} \quad (1)$$

where E_{ideal} is the total energy of the complete crystal and E_{∞} is the total energy of the two separated halves when the spacing between the cleavage planes is infinite.

Ti_5Si_3 crystal belongs to complex hexagonal structure (D8_8), whose stacking density is estimated to be around 71%~76%. The interstitial between the atoms is around 0.16 nm thus it does not

seem likely that interstitution of alloying elements in Ti_5Si_3 crystal is possible. Alloying additives Nb or Cr are mounted in the lattice randomly in a way that substitutes for part of the Ti sites. Many body potential [6] is employed in the total energy calculation. Fig. 1 illustrates the dependence of total energy on the inter-planar spacing of the four low index planes. The horizontal axis is the deviation from the equilibrium inter-planar spacing of the cleavage plane which is in the unit of the equilibrium inter-planar spacing. The vertical axis indicates the calculated total energy and the energy for complete crystal is set as zero. Relaxation was made at each step of the calculation. It can be seen that by increasing the inter-planar spacing, the total energy of the system increases, which makes the reasonable physical sense of fracture. The asymptotic value of the curve gives the ideal cleavage energy for each plane. By comparing the three graphs, it is shown that the cleavage energies of all four calculated cleavage planes are increased by Nb or Cr addition to Ti_5Si_3 .

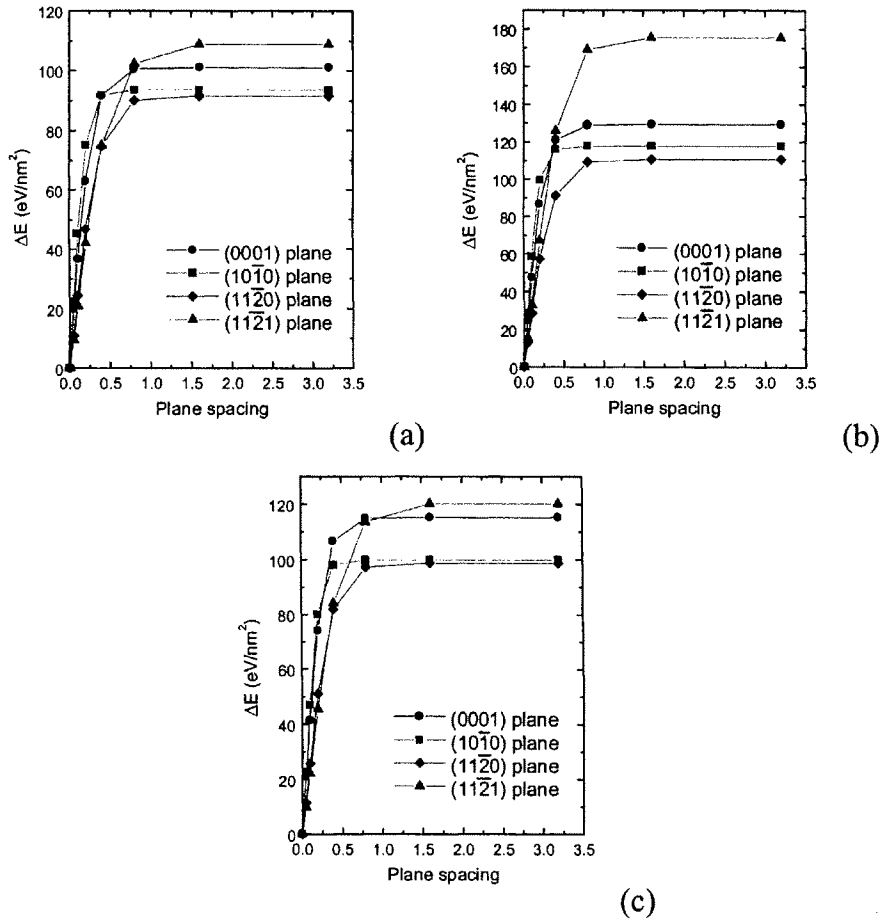


Fig. 1 Calculated total energy as a function of inter-planar spacing (a) Ti_5Si_3 ; (b) Ti_5Si_3 with 2 at. % Nb additive; (c) Ti_5Si_3 with 2 at. % Cr additive.

EXPERIMENTAL PROCEDURE

With Nb or Cr addition to Ti_5Si_3 , two means of alloying are designed : stoichiometric alloying and off-stoichiometric alloying. In stoichiometric alloying, the compositions of alloys are designed to be (in at. %) : $Ti_{62.5-X}M_XSi_{37.5}$ ($M=Nb, Cr$). In other words, Nb or Cr substitutes for a part of the Ti content in the compound's composition while keeping the Si content constant and the ratio of metal elements to silicon as 5 : 3. In off-stoichiometric alloying, the Ti content keeps constant while the Si content is decreased as the Nb or Cr addition is increased. The compositions of the off-stoichiometric alloys are (at. %) : $Ti_{62.5}Si_{37.5-X}M_X$ ($M=Nb, Cr$). Table I lists the nominal compositions of the alloys in this study.

Table I Nominal compositions of the alloys.

Alloy label	Composition (at. %)
Stoichiometric alloys ($Ti_{62.5-X}M_XSi_{37.5}$)	
Ti_5Si_3	Ti-37.5Si
2Nb	Ti-2Nb-37.5Si
5Nb	Ti-5Nb-37.5Si
10Nb	Ti-10Nb-37.5Si
20Nb	Ti-20Nb-37.5Si
1Cr	Ti-1Cr-37.5Si
2Cr	Ti-2Cr-37.5Si
5Cr	Ti-5Cr-37.5Si
Off-stoichiometric alloys ($Ti_{62.5}Si_{37.5-X}M_X$)	
off-5Nb	Ti-32.5Si-5Nb
off-10Nb	Ti-27.5Si-10Nb
off-2Cr	Ti-35.5Si-2Cr

The Ti_5Si_3 based intermetallic compounds were prepared by arc melting pure Ti, Si, Nb and Cr elements under an argon atmosphere in a laboratory furnace. The microstructure and phase constitution of the compounds were characterised by X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX). Elastic moduli of the compounds were determined using the method described in our previous paper [7].

EXPERIMENTAL RESULTS

Stoichiometric alloys

XRD shows that these alloys remain to be monolithic Ti_5Si_3 phase even the Nb content is as high as 20 at. %. The binary Ti_5Si_3 specimen displays an ideal diffraction pattern of the monolithic Ti_5Si_3 phase. The diffraction patterns for the alloyed compounds exhibit the similar pattern yet the peak positions shift to the lower angle, which indicates an expansion in lattice [7].

A needle-like morphology is found in the binary Ti_5Si_3 specimen and Cr alloyed specimens. However, the Nb alloyed specimens are uniform monolithic microstructure where no needles are found. In binary Ti_5Si_3 , the straight needles form a net structure within the grains without making crosses over the grain boundary (Fig. 2(a)). The amount of needles decreases with increasing Cr content in Cr alloyed compounds (Fig. 2(b)-(d)). The needles which are embedded within the grains gradually disappear as the Cr content increases and, only those along the grain boundary which are in the lath form after etching can be found occasionally. The compositions of the needles, determined by EDX spot analysis, show little difference from the matrix composition. Grain boundaries in Cr alloyed specimens are found to be Cr-rich which is about 12 at. % in alloy 5Cr. Table II lists the matrix compositions of the alloys determined by EDX, where it can be seen that Ti_5Si_3 dissolves a large amount of Nb or Cr. Furthermore, if we examine Table II carefully, it can

be found that the Si content in the table for every occasion is around 37.5 at. % which is the stoichiometric content of Ti_5Si_3 compound.

Table II Matrix compositions of the stoichiometric alloys determined by EDX.

Alloy label	Ti_5Si_3	20Nb	1Cr	2Cr	5Cr
Composition (at. %)	Ti 61.2 Si 38.8	Ti 42.5 Nb 21.0 Si 36.5	Ti 62.0 Cr 0.9 Si 37.1	Ti 61.2 Cr 1.3 Si 37.5	Ti 59.8 Cr 2.7 Si 37.5

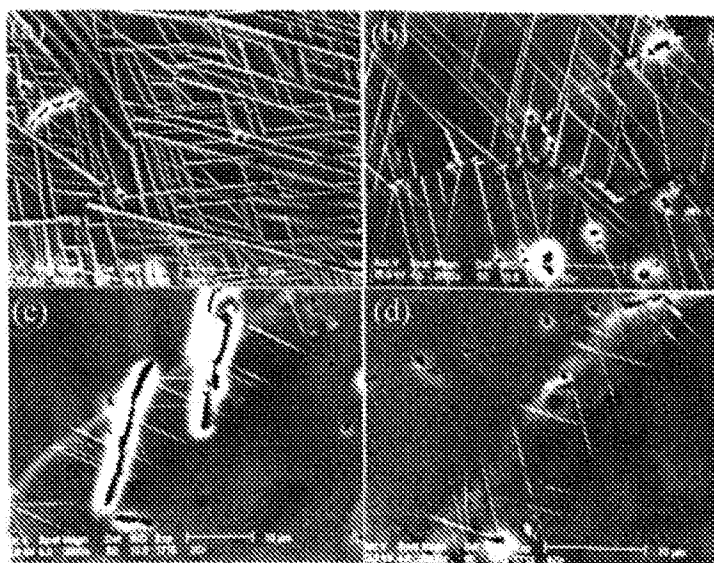


Fig. 2 SEM micrographs of the binary Ti_5Si_3 and Cr alloyed stoichiometric alloys after etching : (a) Ti_5Si_3 , (b) alloy 1Cr, (c) alloy 2Cr and (d) alloy 5Cr.

The elastic modulus of the binary Ti_5Si_3 is measured to be 146.4 GPa and is in good agreement with the 156 GPa obtained by a resonance frequency method reported in literature [1]. The moduli of the Nb or Cr alloyed stoichiometric alloys increase with increasing alloying element content (see Table III) indicating that the Ti_5Si_3 crystal is stiffened, to certain extent, by Nb and Cr additions. This is in agreement with the theoretical calculation.

Table III Measured elastic moduli of the stoichiometric alloys (GPa).

Alloy label	Ti_5Si_3	5Nb	10Nb	20Nb	2Cr	5Cr
E (GPa)	146.4	166.4	177.8	190.8	148.6	162.7

Off-stoichiometric alloys

By increasing the alloying element additions while decreasing the Si content in the compound, the off-stoichiometric alloys are hypereutectic microstructures, as shown in Fig. 3. EDX analysis determines that the primary phase in these alloys is Ti_5Si_3 , which dissolves a certain amount of alloying elements (see Table IV). The content of alloying elements in Ti_5Si_3 phase is related to the amount of alloying element additions to the alloy : the more the addition is, the higher the alloying element content in Ti_5Si_3 is. Furthermore, it can also be noticed that the average Si content of these primary Ti_5Si_3 phases is around 37.5 at. %, which is the stoichiometric Si content in the binary Ti_5Si_3 . The volume percentage of the eutectic area increases as the alloying element content increases.

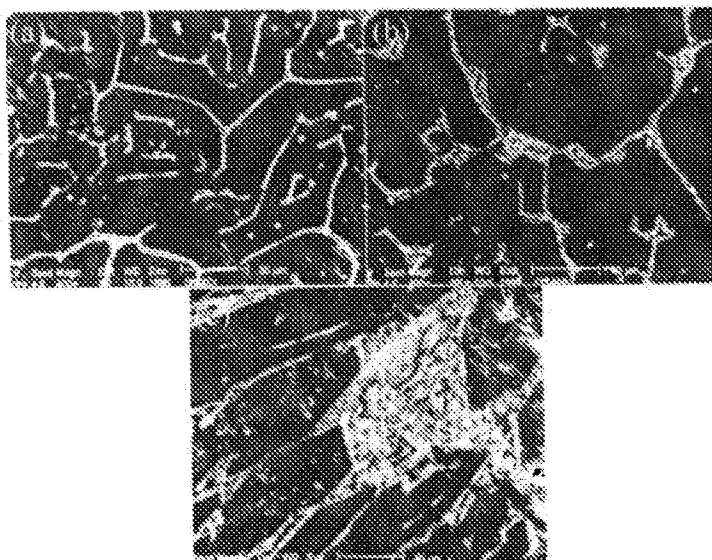


Fig. 3 SEM micrographs of the off-stoichiometric alloys : (a) alloy off-2Cr, (b) alloy off-5Nb and (c) alloy off-10Nb.

Table IV Compositions of the primary Ti_5Si_3 phases in the off-stoichiometric alloys.

Alloy label	off-2Cr	off-5Nb	off-10Nb
Composition (at%)	Ti 60.8 Cr 0.6 Si 38.7	Ti 55.4 Nb 5.4 Si 39.1	Ti 52.7 Nb 8.2 Si 39.1

DISCUSSION

Consulting the binary phase diagrams, it is found that the Ti-Si, Nb-Si and Cr-Si systems are somewhat similar to one another because there exists a M_5Si_3 -type compound (Ti_5Si_3 , Nb_5Si_3 and Cr_5Si_3) in all these systems. The Ti-Nb and Ti-Cr systems, on the other hand, form a fully dissolved solution between the pure elements. Hinted by the information from the binary phase diagrams and the

crystallographic calculation on the interstitials in Ti_5Si_3 crystal [8], the basic idea of "stoichiometric alloying" in this study is that Nb and Cr atoms may partially substitute for Ti atoms in Ti_5Si_3 crystal. Experimental results have revealed that alloying in two different ways (stoichiometric and off-stoichiometric alloying) in this study leads to two different kinds of alloys : the single-phased ones and the multi-phased ones. It has also been shown that Ti_5Si_3 phase can dissolve large amount of Nb or Cr. The stoichiometric alloys are composed of monolithic Ti_5Si_3 phase whose Nb and Cr contents can be as high as 21 at. % and 12 at. % respectively, while the off-stoichiometric alloys contain Nb or Cr dissolved Ti_5Si_3 as the primary phase. Although the designed compositions of the stoichiometric alloys are $\text{Ti}_{62.5-X}\text{M}_X\text{Si}_{37.5}$, the actual compositions of the alloyed Ti_5Si_3 phases determined by EDX can also be described in this way within the reasonable measurement error (see Table II). Rather, even in the off-stoichiometric alloys whose nominal compositions are $\text{Ti}_{62.5}\text{M}_X\text{Si}_{37.5-X}$, the compositions of the primary Ti_5Si_3 phases can still be formulated as $\text{Ti}_{62.5-X}\text{M}_X\text{Si}_{37.5}$ (see Table IV). So the chemical expression for Nb or Cr alloyed Ti_5Si_3 phase can be abbreviated as $(\text{Ti},\text{M})_5\text{Si}_3$ ($\text{M}=\text{Nb}$ or Cr) which means Nb or Cr replaces part of Ti in the compounds.

CONCLUSIONS

Stoichiometric alloying leads to alloys composed of monolithic Ti_5Si_3 phase, while off-stoichiometric alloying leads to hypereutectic alloys with Ti_5Si_3 as the primary phase.

Alloying of Nb and Cr elements in Ti_5Si_3 is substitutional. Nb and Cr substitute for part of Ti in the Ti_5Si_3 crystal while keeping the Si content constant. The maximum observed amount of Nb dissolved in Ti_5Si_3 is 21 at. % and the amount of dissolved Cr is 12 at. %.

Both theoretical calculation and experiment indicate that Nb and Cr additions to the Ti_5Si_3 crystal enhance the bonding between the atoms.

ACKNOWLEDGEMENT

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